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Laboratory Procedure for the Preparation of Solid Waste Related Materials for Anal

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The term solid waste related materials as used here refers to raw refuse such as is delivered to an incinerator or land-fill for disposal, to residues of the incineration process, and to refuse in various stages of composting. The assumption is made that major glass, ceramics and metal components have been removed prior to submission to the laboratory. The three basic operations to be applied to prepare them for detailed analysis are drying, grinding or pulverizing and mixing. The end products of these operations must be so thoroughly homogenized that portions weighing as little as 100-200 mg may be extracted for analysis with full confidence in their being uniform and representative so that any lack of precision or accuracy in the final results cannot be ascribed to the sample.

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LABORATORY PROCEDURE FOR THE PREPARATION OF SOLID WASTE RELATED MATERIALS FOR ANALYSIS

A Solid Waste Research
Open-File Report (RS-03-68-17)

written by
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U.S. ENVIRONMENTAL PROTECTION AGENCY
Office of Research and Monitoring
Solid Waste Research
1971

TABLE OF CONTENTS

DISCUSSION ---------- 1

APPARATUS ---------- 1

SAFETY PRECAUTIONS ---------- 2

PROCEDURE FOR COMBUSTIBLES ---------- 3

- DRYING ---------- 3
- GRINDING IN HAMMERMILL ---------- 4
- FINE GRINDING ---------- 8
- MIXING ---------- 10
- DISCARD ---------- 10

PROCEDURE FOR NON-COMBUSTIBLES ---------- 10

DISCUSSION

The term solid waste related materials as used here refers to raw refuse such as is delivered to an incinerator or land-fill for disposal, to residues of the incineration process, and to refuse in various stages of composting. The assumption is made that major glass, ceramics and metal components have been removed prior to submission to the laboratory. The three basic operations to be applied to prepare them for detailed analysis are drying, grinding or pulverizing and mixing. The end products of these operations must be so thoroughly homogenized that portions weighing as little as 100-200 mg may be extracted for analysis with full confidence in their being uniform and representative so that any lack of precision or accuracy in the final results cannot be ascribed to the sample.

APPARATUS

Basically the apparatus needed to perform the above operations are ovens (preferably mechanical convection) cutting, grinding and pulverizing tools, balances and a mixing device. The following is a listing of apparatus which have been found useful:

1. Ovens: Freas model 114, Thelco model 28
   Blue M model POM-326 FXX
2. Pans for drying
3. Balance - Ohaus-heavy duty solution balance

4. Balance - top loading, 1000 g capacity
5. Desiccator cabinet, large capacity
6. Shears, heavy duty
7. Hatchet
8. Saw
9. Plastic sheet
10. Sample splitter - Gilson - large capacity
11. Hammermill - W-W Grinder - Model F-21-P
12. Wiley mill #3
13. Micromill - Weber Bros. laboratory pulverizing mill
14. Ore pulverizer - Iler
15. Sieves - 10 mesh and 60 mesh
16. Sieve shaker
17. Rotating mixer
18. Miscellaneous gloves, scoops, funnels, containers, brushes, etc.
19. Dust collector - Norblo - Bag type size 48-3, Type BA-2.3

SAFETY PRECAUTIONS

Use gloves if possible when handling refuse (neoprene coated canvas).

Wear some sort of face mask, such as a surgical mask, when preparing samples--especially in finely divided form. It is advisable to wear a transparent plastic face shield while feeding material into the hammermill. Do not use hand to help push material into hammermill past the

feed slot. Use a stick if necessary. Do not open any grinding device while it is running. If any mill clogs turn off motor before clearing apparatus.

PROCEDURE FOR COMBUSTIBLES

The procedures for all types of organic materials are essentially similar, provided that it is borne in mind that compost samples have already been coarsely ground either in a hammer mill or rasping device. It is preferable to dry the materi-l before grinding. If the sample is small (3-5 pounds of moist compost or less of a bulkier material) the drying can be carried out conveniently in a large laboratory oven. If the complete sample is dried an industrial sized oven is most convenient. Where the latter is not available, a preliminary grinding step in a hammer mill is usually necessary.

Drying

1. Small sample in laboratory oven or large sample in industrial oven

1.1 Weigh pan or pans

1.2 Transfer material to pan and reweigh

1.3 Note weight of sample

1.4 Dry in oven at 70-75 C overnight or for 24 hours

1.5 Remove sample and allow to cool, preferably in desiccator, or, if one is not available, covered with aluminum foil

1.6 Reweigh

1.7 Replace in oven for 1-2 hours

1.8 Repeat 1.5 and 1.6.

1.9 If the weight loss is less than 1% of total previous weight loss calculate % moisture from total weight lost.

1.10 If weight loss is over 1% of total previous loss, dry for an additional hour. Continue intermittent drying till the change in calculated moisture is within limit set in 1.9

Example: 0.10% in 10% moisture

0.40% in 40% moisture

Grinding in Hammermill

Procedure	Comments
1. Dried Sample	1. This does not apply to compost or other material previously ground.
1.1 Place sample collection box under mill.	
1.2 Plug lead into power outlet.	
1.3 Open cut off in duct of dust collecting system.	
1.4 Start blower.	
1.5 Oil grinder bearings with engine oil.	1.5 Motor bearings are permanently lubricated.
1.6 Put on personal safety equipment.	
1.7 Start motor.	
1.8 Feed sample into mill.	1.8 If components of sample

1.8 have been segregated by categories, mix these during feeding; tear thick magazines into thinner sections; separate thick wads of paper; remove staples from magazines and cartons and put into container marked discard; place bottle caps and other metal objects into 'discard'; place other non-combustibles (glass, ceramics) not previously removed from sample into 'discard'; cut articles of clothing into smaller sections before feeding into mill; saw or chop large pieces of wood or thick branches into smaller pieces; jeans and work clothing often have metal buttons or

staples which should go into 'discard' container; close metal inlet door after inserting each batch of material

1.9 Turn off grinder motor

1.9 Remove electrical plug from outlet

1.10 Turn off blower

1.11 Clean out grinder and add material to ground sample

1.11 Remove clean out plate at rear of grinder; remove threads wound around shaft between hammers; cut long strings or threads into short pieces; use electric light to illuminate dark areas; scrape off bits of material caught in crevices

1.12 If the sample is appreciably over two pounds, reduce it to about this weight by mixing on a plastic sheet and quartering or passing through a sample splitter. For this purpose,

1.12 The large sized splitter should be used with apertures about 4" wide.

2. Undried Sample

Procedure	Comments
2.1 - 2.11 same as 1.1 - 1.11	2.1 - 2.11 More care must be exercised in cleaning inside of hammer mill since moist material will stick to walls of mill more readily. It is advisable to line sample collection box with plastic film or bag.
2.12 Spread ground product on sheet of plastic and mix rapidly without compacting.	2.12 The sample may be mixed by manipulating the corners and sides of sheet to move the particles from one part to another; a spade is often useful in this operation; compaction of the particles reduces their mobility.
2.13 Quarter the material down to manageable size.	2.13 About 3 pounds is convenient size sample; store remainder in a labeled plastic bag.

2.14 Dry sample as outlined under

"Drying" 1. to 1.10

3. Compost A compost sample as received in the laboratory may be quite wet or appear dry. In either case, the sample should be dried before proceeding any further (unless assurances are received that the sample has been dried). This is carried out as detailed in section 1. - 1.10. Compost usually contains varying quantities of glass and ferrous and non-ferrous metal. If the sample has been relatively finely ground, it is not practical to remove the glass or non-ferrous metal. The ferrous metal can be picked out by combing the sample with a strong magnet. If the sample is quite coarse, much of the glass will be of a size that can be removed without too much difficulty by judicious sieving and the use of a current of air to scatter the lighter organic particles. If the carbon/nitrogen ratio is to be determined identifiable plastic should be removed because it is not degradable. Removal of glass and metal reduces the wear on the Wiley mill knives. After the above preliminary procedures, continue with those in Fine Grinding.

"Fine Grinding"

The material which has been reduced in a hammer mill and dried is now ground in a Wiley mill to pass through a 2mm sieve at least and preferably through a 1 mm sieve.

	Procedure	Comments
1.1	Put 2 mm sieve into Wiley mill and close mill.	
1.2	Open cut off in dust collection duct.	
1.3	Position container under delivery spout.	1.3 A standard Mason jar can be screwed directly into the spout; if the ground sample is caught in a different type container, it is advisable to provide it with a covering with a hole in it through which the spout is inserted.
1.4	Replace 2 mm sieve with 1 mm sieve and regrind the sample.	1.4 The finer the grind, the more uniform the final sample can be made.
1.5	Brush out all inside surfaces of the mill into a separate container.	1.5 This includes the hopper, the walls, the screen, the spout; remove as much as possible of the material wedged between the knives and their housing.
1.6	Put this material through a micro mill.	1.6 On the Weber mill the 0.05" or 0.10" screen may be used. For

1.6 small samples substitute a long narrow plastic bag for the canvas bag provided.

1.7 Add the product to the main sample and mix.

Mixing

The final mixing or homogenization is accomplished by transferring the sample to a suitable container which it will fill to a point not over half full. The container is closed tightly, positioned in the rotating mixer and allowed to mix for not less than one hour, preferably two hours. The mixed sample may be reduced in size if desired by means of a sample splitter or by quartering.

Discard

Weigh all metal, ceramics, plastic and glass removed during processing. This information will be used later in calculations.

PROCEDURE FOR NON-COMBUSTIBLES

	Procedure	Comments
1.	Incinerator residues are usually wet when received and are dried at 100-105 C.	1. The temperature or time of drying are not critical. A 10 pound sample or smaller is usually enough to dry.

1.1	Weigh the dried sample	1.1	If the moisture content is to be determined, this value is of course obtained in the routine procedure.
1.2	Work the sample over with a strong magnet to remove ferrous metal and magnetic iron oxide.	1.2	If the magnet is enclosed in a piece of cloth, it will be easy to remove the magnetic material clinging to it by separating the cloth from the magnet. The removal is otherwise quite difficult to effect.
1.3	Sieve the sample through a 1/4" mesh sieve and pick out glass and ceramics.	1.3	While the pulverizer will grind this material, the removal of ingredients not contributing to the B.T.U. value will enrich a sample whose B.T.U. value is low under the best conditions.
1.4	Check the oil level in the pulverizer. The oil cup should be quite full.	1.4	The gears run in a bath of oil. We are using regular engine oil. Be careful not to get any oil into sample compartment.
1.5	Adjust the movable pulverizer plate to give a maximum size of about 10 mesh.	1.5	This is accomplished by rotating the wheel with the holes in the rim (just ahead of the fly wheel)

		1.5	clockwise to give a coarse product and counter-clockwise to give a fine product.
1.6	Put the sample through the pulverizer.	1.6	When restarting the motor after emptying the product pan, it is advisable to empty the grinding chamber. Starting the motor against a load may cause burnout.
1.7	Screen the ground material through 10 mesh and 60 mesh sieves. The material through the 60 mesh is final sample. The remainder is reground with the plates placed together with minimum clearance.	1.7	It may be necessary to make multiple passes through the pulverizer to grind the whole sample through the 60 mesh sieve. The coarse material will often disclose bits of metal which have been burnished during the first passage through the pulverizer. These may be picked out and added to the discard (see procedure and comments 1.2 and 1.3)
1.8	Mix as indicated under "Mixing".		

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